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PATENT

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on December 9, 2002


Signature

Applicant : Keiko Matsubara, et al.
Application No. : 09/672,287
Filed : September 28, 2000
Title : NEGATIVE ACTIVE MATERIAL FOR RECHARGEABLE
LITHIUM BATTERY ELECTRODE FOR RECHARGEABLE
LITHIUM BATTERY, RECHARGEABLE LITHIUM
BATTERY AND METHOD OF PREPARING NEGATIVE
ACTIVE MATERIAL FOR RECHARGEABLE LITHIUM
SECONDARY BATTERY

Grp./Div. : 1745
Examiner : Dah Wei D. Yuan

Docket No. : 40589/DBP/Y35

RECEIVED
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TC 1700

RULE 132 DECLARATION

Assistant Commissioner for Patents
Washington, D.C. 20231 November 26, 2002

Post Office Box 7068
Pasadena, CA 91109-7068

Commissioner:

I, Keiko Matsubara, hereby declare that:

1. I was a research assistant at the department of Physics of Nihon University from 1982 to 1991. I received a Ph.D. in Physics from Nihon University in 1992. I was a full-time instructor at the department of Electrical Engineering of the College of Science & Technology of Nihon University from 1992 to 1998.

2. At Nihon University, I have studied the physical properties of graphite intercalation compounds, and also evaluated the crystal structure of graphite powder used as an anode material in Li ion battery. From 1998 to present, I am in process of developing next-generation anode materials for Lithium ion battery at Samsung Yokohama Research Institute. I have focused on the composite material of graphite and metals capable to be made into an alloy with lithium, and I have investigated the relation between the morphologic feature of a compound particle and its battery characteristic specifically. I consider myself to be an expert in the field of anode materials for Li ion batteries.

3. I have reviewed U.S. Application No. 09/672,287. I understand that the application claims, *inter alia*, a negative active material for a rechargeable lithium battery comprising a particle-agglomerated product comprising a carbonaceous material and an amorphous metal compound, the carbonaceous material being a material into or from which lithium is intercalated or deintercalated, and the amorphous metal compound being able to make an alloy with lithium and including one or more metals selected from the group consisting of Sn, Ag, Fe, Pd, Pb, Al, Si, In, Ni, Co, An and Cd.

4. The particle-agglomerated product produced as described in U.S. Patent Application No. 09/672,287 is a composite product in which an amorphous metal compound and carbonaceous material are integrated through coating and agglomerating processes using an agglomerating device and calcination process. The resulting particle-agglomerated product is in the form of a powder.

5. I have reviewed U.S. Patent No. 6,004,695 to Goda et al. Goda describes a method whereby an amorphous tin-based composite oxide (a negative active material), a flake binder (a conductive agent), a polyvinylidene fluoride dispersion (a binder), carboxymethyl cellulose (a binder), and lithium acetate (an additive) are kneaded in water to prepare a slurry. The slurry is coated onto copper foil and dried, and then the coated copper foil is compressed to prepare a negative electrode.

6. I prepared a dried slurry product in accordance with the method described in Goda. I made the slurry by mixing 15 wt% tin acetate, 75 wt% natural graphite, 10 wt% polyvinylidene fluoride (PVdF) and 100 wt% water. The amounts of natural graphite and tin acetate used were the same as the amounts used in Example 1 of U.S. Patent Application No. 09/672,287. However, because Example 1 of U.S. Patent Application No. 09/672,287 is directed to a method for obtaining an active material, and not a method for obtaining a slurry, it was necessary for me to increase the amount of water in the described experiment relative to the amount used in Example 1 in order to prepare a slurry. After 1 hour of kneading, the slurry was coated on a copper foil and dried in a vacuum oven. U.S. Patent No. 6,004,695 does not describe a drying temperature, so the coated slurry was dried at 120(degrees)C, which is the general drying temperature used for slurries containing PVdF.


7. Appended hereto are photographs of (a) natural graphite having a flake shape; (b) the dried slurry product prepared according to the same kneading method as in Goda; and (c) the particle-agglomerated product of U.S. Patent Application No. 09/672,287. Photograph (c) is the same as FIG. 4 of U.S. Patent Application No. 09/672,287. As shown in photograph (b), the dried products of the tin acetate solution were deposited on the surface of the natural graphite, and the natural graphite maintained a tin flake shape, indicating that a particle-agglomerated product was not produced. As shown in photograph (c), the natural graphite produced according to U.S. Patent Application No. 09/672,287 was agglomerated and stacked in multiple layers, with tin oxides present between the multiple layers. In contrast, with the method of Goda, the dried products of the tin acetate solution were only on the surface of the natural graphite.

8. In my opinion, as an expert in the field of anode materials for Li ion batteries, the Goda patent does not describe a negative active material in particle-agglomerated form, as described and claimed in U.S. Patent Application No. 09/672,287.

9. I declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are

punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

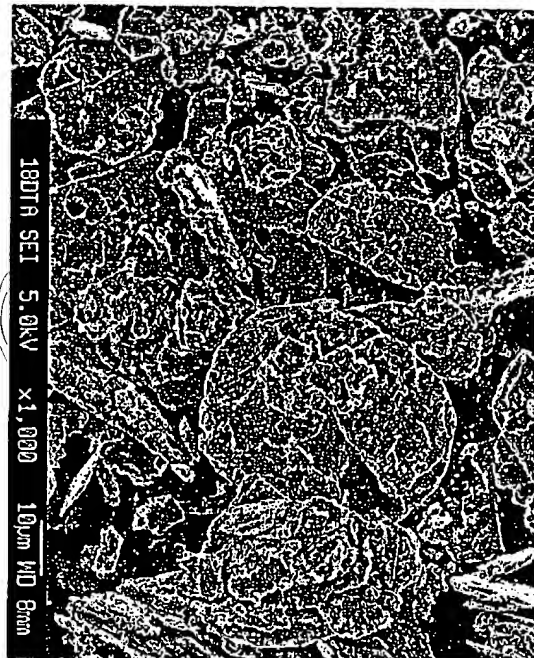
Date 2002/11/26

By 
Dr. Keiko Matsubara

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(a)



(b)



(c)